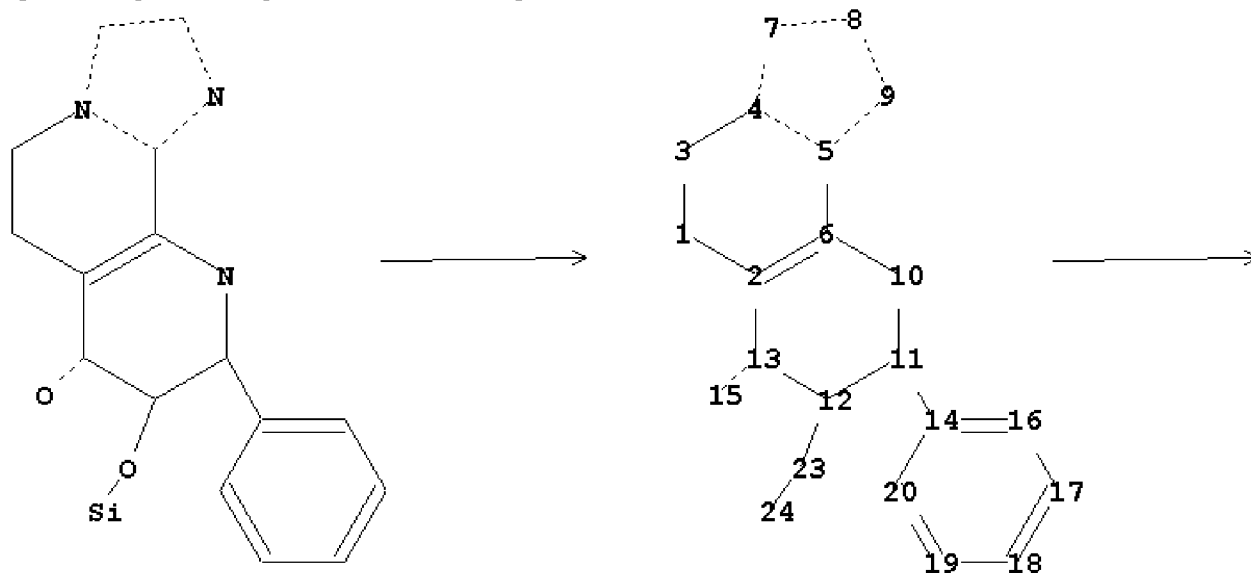


=>

Uploading C:\Program Files\Stnexp\Queries\10550691.str



chain nodes :

15 23 24

ring nodes :

1 2 3 4 5 6 7 8 9 10 11 12 13 14 16 17 18 19 20

chain bonds :

11-14 12-23 13-15 23-24

ring bonds :

1-3 1-2 2-6 2-13 3-4 4-5 4-7 5-6 5-9 6-10 7-8 8-9 10-11 11-12 12-13
14-16 14-20 16-17 17-18 18-19 19-20

exact/norm bonds :

1-3 1-2 2-6 2-13 3-4 4-5 4-7 5-6 5-9 6-10 7-8 8-9 10-11 11-12 12-13
12-23 13-15

exact bonds :

11-14 23-24

normalized bonds :

14-16 14-20 16-17 17-18 18-19 19-20

Match level :

1:CLASS 2:CLASS 3:CLASS 4:CLASS 5:CLASS 6:CLASS 7:CLASS 8:CLASS 9:CLASS
10:CLASS 11:CLASS 12:CLASS 13:CLASS 14:CLASS 15:CLASS 16:CLASS 17:CLASS
18:CLASS 19:CLASS 20:CLASS 23:CLASS 24:CLASS

L1 STRUCTURE UPLOADED

=> s 11

SAMPLE SEARCH INITIATED 20:29:03 FILE 'CASREACT'

SCREENING COMPLETE - 0 REACTIONS TO VERIFY FROM 0 DOCUMENTS

100.0% DONE 0 VERIFIED 0 HIT RXNS 0 DOCS

SEARCH TIME: 00.00.01

FULL FILE PROJECTIONS: ONLINE **COMPLETE**
BATCH **COMPLETE**

PROJECTED VERIFICATIONS: 0 TO 0
PROJECTED ANSWERS: 0 TO 0

L2 0 SEA SSS SAM L1 (0 REACTIONS)

=> s l1 ful

FULL SEARCH INITIATED 20:29:20 FILE 'CASREACT'

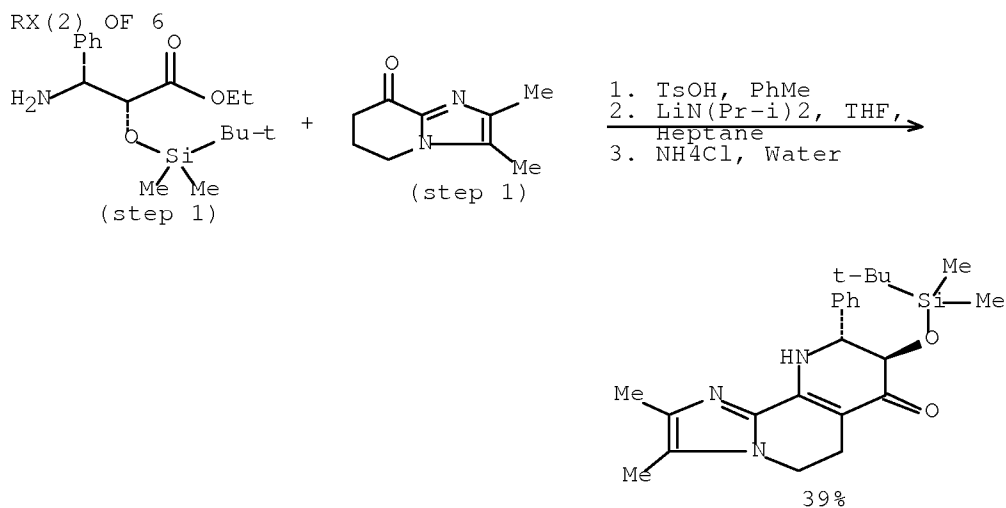
SCREENING COMPLETE - 232 REACTIONS TO VERIFY FROM 17 DOCUMENTS

100.0% DONE 232 VERIFIED 7 HIT RXNS 2 DOCS
SEARCH TIME: 00.00.01

L3 2 SEA SSS FUL L1 (7 REACTIONS)

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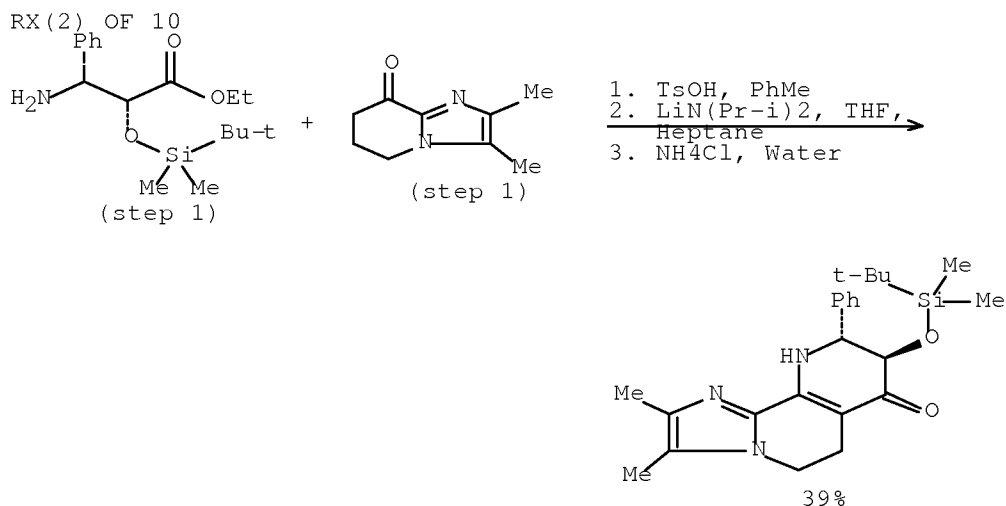
L3 ANSWER 1 OF 2 CASREACT COPYRIGHT 2008 ACS on STN



REF: PCT Int. Appl., 2004087718, 14 Oct 2004

CON: STAGE(1) reflux
STAGE(2) reflux -> -15 deg C; 1 hour, -15 deg C; 30 minutes,
-15 deg C -> -5 deg C

L3 ANSWER 2 OF 2 CASREACT COPYRIGHT 2008 ACS on STN



REF: PCT Int. Appl., 2004056362, 08 Jul 2004

CON: STAGE(1) reflux
 STAGE(2) 1.5 hours, room temperature
 STAGE(3) -5 deg C

<http://www.cas.org/legal/infopolicy.html>

=> s (77 and 1955 and 55)/so
 198131 77/SO
 89090 1955/SO
 268698 55/SO

L2 10 (77 AND 1955 AND 55)/SO

=> d ti

L2 ANSWER 1 OF 10 CAPLUS COPYRIGHT 2008 ACS on STN
 TI The serum calcium content during androgen therapy of female genital carcinoma

=> d ti 2-10

L2 ANSWER 2 OF 10 CAPLUS COPYRIGHT 2008 ACS on STN
 TI Tiglic and angelic acids

L2 ANSWER 3 OF 10 CAPLUS COPYRIGHT 2008 ACS on STN
 TI Liquid-vapor phase equilibrium in solutions of oxygen and nitrogen at pressures below one atmosphere

L2 ANSWER 4 OF 10 CAPLUS COPYRIGHT 2008 ACS on STN
 TI The preparation of organometallic and organometalloidal compounds by the diazoalkane method

L2 ANSWER 5 OF 10 CAPLUS COPYRIGHT 2008 ACS on STN
 TI Reactivities of aromatic hydrocarbons toward methyl radicals

L2 ANSWER 6 OF 10 CAPLUS COPYRIGHT 2008 ACS on STN
 TI The configuration and reactivity of 9-substituted decalins

L2 ANSWER 7 OF 10 CAPLUS COPYRIGHT 2008 ACS on STN
 TI Some dehydrogenation products of α -carotene, β -carotene, and cryptoxanthin

L2 ANSWER 8 OF 10 CAPLUS COPYRIGHT 2008 ACS on STN
 TI William Lloyd Evans, December 22, 1870-October 18, 1954

L2 ANSWER 9 OF 10 CAPLUS COPYRIGHT 2008 ACS on STN
 TI Photosynthesis by isolated chloroplasts. III. Evidence for complete photosynthesis

L2 ANSWER 10 OF 10 CAPLUS COPYRIGHT 2008 ACS on STN
 TI The mechanism of enzyme action. LIX. A relation between the structure of mold pigments and their interaction with enzymes

=> d abs 7

L2 ANSWER 7 OF 10 CAPLUS COPYRIGHT 2008 ACS on STN
 AB By treatment of α -carotene (I), β -carotene (II), and cryptoxanthin (C₁₀H₅₅OH) (III) with N-bromosuccinimide (IV) and (in part) with AcNHBr, 3 new crystalline pigments, the "dehydrocarotenes I, II, and III" have been isolated, besides dehydro- β -carotene (V), bisdehydro- β -carotene (VI), and anhydroeschscholtzxanthin (VII). Dehydrocarotene I (VIII) was obtained only from I, II (IX) from II, and III (X) from I and II. Their spectral curves were characterized by the lack of fine structure in the visible region. Upon treatment of VIII, IX, and X [or of deoxylutein I (XI)] with Et₂O-BF₃ and subsequent chromatog. resolution, fine structure appeared in the spectrum of the main product. Some structural, stereochem., and spectral features of these compds. are discussed. Pure crystalline I (500 mg.) in 50-mg. portions refluxed 6 h. over a 200-w. elec. bulb with 33 mg. IV, 15 cc. CCl₄, and 75 mg. NaHCO₃ each, the mixture diluted with hexane to 25 cc., and the solution from 10 similar runs combined and developed with hexane + 8% Me₂CO on five 30 + 8-cm. lime-celite columns gave the following zones (width of the zones given in mm.): 2, pale brown; 125, interzone; 15, pink (all-trans-VI); 11, interzone; 13, purple (all-trans-VII); 14, interzone; 23, pink (a cis-VI); 5, interzone; 9, light purple (a cis-VII); 83, two pale pink zones (and interzones); and an orange filtrate. The all-trans-VI and all-trans-VII zones cut out, eluted, transferred into hexane with H₂O, the solns. dried, combined with the identical ones from the other columns, the material rechromatographed in the same manner, the solns. evaporated, both residues dissolved in a little warm C₆H₆, the solution concentrated in a N stream, and the concentrate diluted dropwise with MeOH gave the crystalline solids. The combined 5 chromatog. filtrates washed Me₂CO-free, dried, concentrated to 500 cc., and developed with hexane on 10 columns gave the following zones: 50, empty; 9, pink (X); 21, interzone; 20, orange (unidentified); 10, interzone; 19, yellow (unidentified); 49, interzone; 39, orange (VIII); 35, pale yellow (unidentified); 40, yellow (unchanged I); 8, empty. X and VIII were rechromatographed in the same manner on MgO, the hexane eluates evaporated, and the crystallized residues recrystd. from C₆H₆-MeOH. The all-trans-VI, dark purple, long, quadrangular plates moderately glittering, m. 199-201°, did not sep. on the column from a sample from II. The all-trans-VII, very dark purple crystals forming parallel twins and bulky conglomerates, m. 211°, showed an epiphasic partition behavior. X, deep red, quadrangular (rhombic), faintly glittering plates, m. 191-2.5° (from C₆H₆-MeOH), was markedly less soluble in hexane than II, was insol. in MeOH, and showed an epiphasic

partition behavior; when developed with hexane + 2-3% Me₂CO on lime-celite, it was adsorbed between the II and the V. VIII, brownish red, glittering plates, m. 188-90° (from C₆H₆-MeOH), hydrogenated in cyclohexane and glacial AcOH over PtO₂ showed 11.4 (11.3) double bonds. The spectrum of VIII in hexane remained essentially unchanged upon shaking 4 h. with 20% KOH in MeOH or upon refluxing 2 h. with alc. NaOEt in C₆H₆. VIII was less soluble than I; it was insol. in MeOH and showed an epiphasic partition behavior; when developed with hexane on lime-celite, it appeared between I and II. VIII (6 mg.) in 50 cc. hexane illuminated 0.5 h. in the presence of 0.08 mg. iodine and developed with hexane on lime-celite gave in the following top-to-bottom sequence: neo U, neo V, unchanged all-trans-VIII, neo A, and neo B, all separated by colorless interzones; these compds. were obtained in the ratio 7.5:1.5:67.5:6.0:17.5; after 45 min. refluxing the ratio was 1.5:0:90.0:5.0:3.5; each cis isomer yielded upon iodine catalysis and chromatog. the all-trans-VIII. I (50 mg.) in 5 cc. CCl₄ treated with 15 mg. AcNHBr in 2 cc. Et₂O, the Et₂O evaporated, and the residue chromatographed gave 35% recovered I, 0.25% VI, 3.5% VIII, and 0.21% X. I (50 mg.) and 42 mg. o-C₆H₄(CO)₂NBr in 75 mL. CCl₄ refluxed 30 min. gave 25% recovered I, trace of VI, 3.8% VIII, and trace of X. VIII (12 mg.) in 12 cc. CHCl₃ shaken vigorously 1 min. with 1.2 cc. Et₂O-BF₃, the mixture treated with some ice-cold H₂O, and the organic layer diluted with hexane, washed acid-free, dried, and developed with hexane + 5% Me₂CO on limecelite gave the following zones: 41, two weak yellow zones and interzones; 20, light orange (main product); 132, several light yellow zones (among them a cis form of the main product); and a fluorescent filtrate. The 20-mm. zone rechromatographed, the hexane solution dried, evaporated, and the residue crystallized from C₆H₆-95% MeOH gave 2.4 mg. C₄₀H₅₆, m. 171-2°; a hexane solution mixed with 1 volume 20% KOH in MeOH and refluxed 2 h. did not show any change of the visual spectrum and of chromatog. homogeneity; the compound was more soluble in C₆H₆ than either II or VIII, was very sparingly soluble in MeOH, and showed an epiphasic partition behavior. VIII exhibited a provitamin A potency of about 15% that of II, or about 1/3 that of I; on the basis of the available data VIII is regarded as 3,4-dehydro-I. II (ten 100-mg. portions) refluxed 2 h. with IV and the reaction mixture developed with hexane + 2% Me₂CO on lime-celite gave the following zones: 7, brownish; 70, V, VI, VII, and some cis forms separated by interzones; 33, interzone; 9, faint pink; 8, interzone; 13, pink (X); 14, orange; 12, interzone; 25, orange (IX); 13, yellowish orange; 6, interzone; 37, II; 5, interzone; 25, yellow. The 3 components in the 70-mm. zone separated, rechromatographed, and crystallized yielded 20 mg. V, 14.5 mg. VI, and 6.5 mg. VII. IX and X rechromatographed on 3:1 MgO-celite, developed with 9:1 hexane-Me₂CO, the solution evaporated, and the residue dissolved in C₆H₆ and recrystd. by the addition of MeOH gave (from 1 g. II) 14 mg. IX, m. 186-7.5°, and 2.4 mg. X, m. 191-2°; IX formed deep orange-red, thick hexagonal plates. The spectrum of IX was not altered by refluxing in C₆H₆ with NaOEt in EtOH during 2 h.; it was less soluble in hexane than II and was epiphasic in the partition test; it was adsorbed on lime-celite above the II. IX isomerized in the presence of iodine gave neo U-IX-neo A-neo B in the ratio 22:48.5:26.5:3. IX (16 mg.) treated with Et₂O-BF₃, the mixture developed with hexane +8% Me₂CO, and the main zone rechromatographed and recrystd. from C₆H₆-98% MeOH yielded 1 mg. product, m. 163.5-4.5°. XI (7 mg.) treated with Et₂O.BF₃ in the usual manner and chromatographed on lime-CaCO₃-celite with 9:1 hexane-Me₂CO gave 2 main zones having roughly the location of I and II. II (50 mg.) in 5 cc. CCl₄ refluxed 20 min. with 15 mg. AcNHBr in 2 cc. Et₂O, and the combined yields of 20 such runs developed with hexane +8% Me₂CO on lime-celite yielded 21% recovered II, 1.6% V, 2.8% neo-A V (m. 159-61°), and 2.1% VI. III (20 mg.) (ex persimmons) in 6 cc. CCl₄ treated with 13 mg. IV and 30 mg. NaHCO₃, the mixture refluxed 45 min., diluted with hexane, developed with hexane +8% Me₂CO on lime-celite, and the corresponding zones from 8 such runs combined and worked up in the usual manner gave 0.73% VI, 0.93% VII, a small amount (0.4%) of X, and a new deep red product which was void of fine structure in the UV, was essentially

epiphasic in the partition test, and was adsorbed on lime-celite above VI. III treated similarly with AcNHBr yielded 1.4% VI, 0.88% VII, and the new product. The UV absorption spectra of all-trans-VIII, all-trans-IX, all-trans-X, of the crystalline compds. from VIII and IX with Et₂O.BF₃, XI, and of the neo U, neo A, and neo B cis forms of IX are recorded.

=> d abs fbib hitstr 7

L2 ANSWER 7 OF 10 CAPLUS COPYRIGHT 2008 ACS on STN

AB By treatment of α -carotene (I), β -carotene (II), and cryptoxanthin (C₁₀H₅₅OH) (III) with N-bromosuccinimide (IV) and (in part) with AcNHBr, 3 new crystalline pigments, the "dehydrocarotenes I, II, and III" have been isolated, besides dehydro- β -carotene (V), bisdehydro- β -carotene (VI), and anhydroeschscholtzxanthin (VII). Dehydrocarotene I (VIII) was obtained only from I, II (IX) from II, and III (X) from I and II. Their spectral curves were characterized by the lack of fine structure in the visible region. Upon treatment of VIII, IX, and X [or of deoxylutein I (XI)] with Et₂O-BF₃ and subsequent chromatog. resolution, fine structure appeared in the spectrum of the main product. Some structural, stereochem., and spectral features of these compds. are discussed. Pure crystalline I (500 mg.) in 50-mg. portions refluxed 6 h. over a 200-w. elec. bulb with 33 mg. IV, 15 cc. CCl₄, and 75 mg. NaHCO₃ each, the mixture diluted with hexane to 25 cc., and the solution from 10 similar runs combined and developed with hexane + 8% Me₂CO on five 30 x 8-cm. lime-celite columns gave the following zones (width of the zones given in mm.): 2, pale brown; 125, interzone; 15, pink (all-trans-VI); 11, interzone; 13, purple (all-trans-VII); 14, interzone; 23, pink (a cis-VI); 5, interzone; 9, light purple (a cis-VII); 83, two pale pink zones (and interzones); and an orange filtrate. The all-trans-VI and all-trans-VII zones cut out, eluted, transferred into hexane with H₂O, the solns. dried, combined with the identical ones from the other columns, the material rechromatographed in the same manner, the solns. evaporated, both residues dissolved in a little warm C₆H₆, the solution concentrated in a N stream, and the concentrate diluted dropwise with MeOH gave the crystalline solids. The combined 5 chromatog. filtrates washed Me₂CO-free, dried, concentrated to 500 cc., and developed with hexane on 10 columns gave the following zones: 50, empty; 9, pink (X); 21, interzone; 20, orange (unidentified); 10, interzone; 19, yellow (unidentified); 49, interzone; 39, orange (VIII); 35, pale yellow (unidentified); 40, yellow (unchanged I); 8, empty. X and VIII were rechromatographed in the same manner on MgO, the hexane eluates evaporated, and the crystallized residues recrystd. from C₆H₆-MeOH. The all-trans-VI, dark purple, long, quadrangular plates moderately glittering, m. 199-201°, did not sep. on the column from a sample from II. The all-trans-VII, very dark purple crystals forming parallel twins and bulky conglomerates, m. 211°, showed an epiphasic partition behavior. X, deep red, quadrangular (rhombic), faintly glittering plates, m. 191-2.5° (from C₆H₆-MeOH), was markedly less soluble in hexane than II, was insol. in MeOH, and showed an epiphasic partition behavior; when developed with hexane + 2-3% Me₂CO on lime-celite, it was adsorbed between the II and the V. VIII, brownish red, glittering plates, m. 188-90° (from C₆H₆-MeOH), hydrogenated in cyclohexane and glacial AcOH over PtO₂ showed 11.4 (11.3) double bonds. The spectrum of VIII in hexane remained essentially unchanged upon shaking 4 h. with 20% KOH in MeOH or upon refluxing 2 h. with alc. NaOEt in C₆H₆. VIII was less soluble than I; it was insol. in MeOH and showed an epiphasic partition behavior; when developed with hexane on lime-celite, it appeared between I and II. VIII (6 mg.) in 50 cc. hexane illuminated 0.5 h. in the presence of 0.08 mg. iodine and developed with hexane on lime-celite gave in the following top-to-bottom sequence: neo U, neo V, unchanged all-trans-VIII, neo A, and neo B, all separated by colorless

interzones; these compds. were obtained in the ratio 7.5:1.5:67.5:6.0:17.5; after 45 min. refluxing the ratio was 1.5:0:90.0:5.0:3.5; each cis isomer yielded upon iodine catalysis and chromatog. the all-trans-VIII. I (50 mg.) in 5 cc. CCl₄ treated with 15 mg. AcNHBr in 2 cc. Et₂O, the Et₂O evaporated, and the residue chromatographed gave 35% recovered I, 0.25% VI, 3.5% VIII, and 0.21% X. I (50 mg.) and 42 mg. o-C₆H₄(CO)₂NBr in 75 mL. CCl₄ refluxed 30 min. gave 25% recovered I, trace of VI, 3.8% VIII, and trace of X. VIII (12 mg.) in 12 cc. CHCl₃ shaken vigorously 1 min. with 1.2 cc. Et₂O-BF₃, the mixture treated with some ice-cold H₂O, and the organic layer diluted with hexane, washed acid-free, dried, and developed with hexane + 5% Me₂CO on limecelite gave the following zones: 41, two weak yellow zones and interzones; 20, light orange (main product); 132, several light yellow zones (among them a cis form of the main product); and a fluorescent filtrate. The 20-mm. zone rechromatographed, the hexane solution dried, evaporated, and the residue crystallized from C₆H₆-95% MeOH gave 2.4 mg. C₄₀H₅₆, m. 171-2°; a hexane solution mixed with 1 volume 20% KOH in MeOH and refluxed 2 h. did not show any change of the visual spectrum and of chromatog. homogeneity; the compound was more soluble in C₆H₆ than either II or VIII, was very sparingly soluble in MeOH, and showed an epiphasic partition behavior. VIII exhibited a provitamin A potency of about 15% that of II, or about 1/3 that of I; on the basis of the available data VIII is regarded as 3,4-dehydro-I. II (ten 100-mg. portions) refluxed 2 h. with IV and the reaction mixture developed with hexane + 2% Me₂CO on lime-celite gave the following zones: 7, brownish; 70, V, VI, VII, and some cis forms separated by interzones; 33, interzone; 9, faint pink; 8, interzone; 13, pink (X); 14, orange; 12, interzone; 25, orange (IX); 13, yellowish orange; 6, interzone; 37, II; 5, interzone; 25, yellow. The 3 components in the 70-mm. zone separated, rechromatographed, and crystallized yielded 20 mg. V, 14.5 mg. VI, and 6.5 mg. VII. IX and X rechromatographed on 3:1 MgO-celite, developed with 9:1 hexane-Me₂CO, the solution evaporated, and the residue dissolved in C₆H₆ and recrystd. by the addition of MeOH gave (from 1 g. II) 14 mg. IX, m. 186-7.5°, and 2.4 mg. X, m. 191-2°; IX formed deep orange-red, thick hexagonal plates. The spectrum of IX was not altered by refluxing in C₆H₆ with NaOEt in EtOH during 2 h.; it was less soluble in hexane than II and was epiphasic in the partition test; it was adsorbed on lime-celite above the II. IX isomerized in the presence of iodine gave neo U-IX-neo A-neo B in the ratio 22:48.5:26.5:3. IX (16 mg.) treated with Et₂O-BF₃, the mixture developed with hexane +8% Me₂CO, and the main zone rechromatographed and recrystd. from C₆H₆-98% MeOH yielded 1 mg. product, m. 163.5-4.5°. XI (7 mg.) treated with Et₂O.BF₃ in the usual manner and chromatographed on lime-CaCO₃-celite with 9:1 hexane-Me₂CO gave 2 main zones having roughly the location of I and II. II (50 mg.) in 5 cc. CCl₄ refluxed 20 min. with 15 mg. AcNHBr in 2 cc. Et₂O, and the combined yields of 20 such runs developed with hexane +8% Me₂CO on lime-celite yielded 21% recovered II, 1.6% V, 2.8% neo-A V (m. 159-61°), and 2.1% VI. III (20 mg.) (ex persimmons) in 6 cc. CCl₄ treated with 13 mg. IV and 30 mg. NaHCO₃, the mixture refluxed 45 min., diluted with hexane, developed with hexane +8% Me₂CO on lime-celite, and the corresponding zones from 8 such runs combined and worked up in the usual manner gave 0.73% VI, 0.93% VII, a small amount (0.4%) of X, and a new deep red product which was void of fine structure in the UV, was essentially epiphasic in the partition test, and was adsorbed on lime-celite above VI. III treated similarly with AcNHBr yielded 1.4% VI, 0.88% VII, and the new product. The UV absorption spectra of all-trans-VIII, all-trans-IX, all-trans-X, of the crystalline compds. from VIII and IX with Et₂O.BF₃, XI, and of the neo U, neo A, and neo B cis forms of IX are recorded.

AN 1956:4573 CAPLUS Full-text

DN 50:4573

OREF 50:894g-i,895a-i,896a-d

TI Some dehydrogenation products of α -carotene, β -carotene, and cryptoxanthin

AU Karmakar, G.; Zechmeister, L.

CS California Inst. of Technol., Pasadena
SO Journal of the American Chemical Society (1955), 77,
55-60
CODEN: JACSAT; ISSN: 0002-7863
DT Journal
LA Unavailable

<http://www.cas.org/legal/infopolicy.html>

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73270 1953/SO
222910 75/SO
664454 18/SO
1410 4493/SO
L4 0 (1953 AND 75 AND 18 AND 4493)/SO

=> s (1953 and 75 and 4493)/so
73270 1953/SO
222910 75/SO
1410 4493/SO
L5 1 (1953 AND 75 AND 4493)/SO

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L5 ANSWER 1 OF 1 CAPLUS COPYRIGHT 2008 ACS on STN

AB β -Carotene (I) refluxed with N-bromosuccinimide (II) yielded a complicated pigment mixture from which dehydro- β -carotene, C₄₀H₅₄ (III), bisdehydro- β -carotene, C₄₀H₅₂ (IV), and a pigment very probably identical with anhydroeschscholtzianthin, C₄₀H₅₀ (V), were obtained in pure state. I (200 mg.) in 60 cc. CCl₄ and 130 mg. II refluxed 6 hrs., the solution cooled, filtered, concentrated in vacuo to 20 cc., diluted with 80 cc. hexane, adsorbed on a 27 + 8 cm. column of 2:1 lime-celite, and eluted with hexane containing 3% Me₂CO gave a brownish red, several red and orange zones, and a yellow-orange zone of 50 mg. unchanged I. The eluate from the red and orange zones transferred into hexane and evaporated completely, the crystalline pigment mixture transferred with the min. amount of C₆H₆ into a small centrifuge tube, the solution treated dropwise at 50° with MeOH with stirring, and the resulting crystals (28 mg.) centrifuged off, dissolved in 150 cc. hexane at 20°, chromatographed on a 27 + 8 cm. column, and eluted with 1:3 C₆H₆-hexane gave the following zones (compound, color, width in mm., and, in parentheses, the ultraviolet absorption maximum in m μ given): all-trans-dehydro- β -carotene (VI), orange-red 25 (504, 474); all-trans-IV, reddish purple, 50 (522, 490); V, purple, 25 (534, 499); neodehydro- β -carotene A (VII), orange, 15 (500, 469); cis-IV, orange-red, 10 (518, 489); neodehydro- β -carotene D, orange, 5 (494, 461); cis-IV, orange, 10 (515, 484); cis-III, yellow-orange, 5 (491, 460); cis-IV, pale orange, 10 (510, 481). The dried C₆H₆-hexane solution of III evaporated and the residue recrystd. from C₆H₆-MeOH yielded 3-4 mg. III, m. 192°. The fraction containing the IV evaporated, the residue dissolved in C₆H₆, and the solution cautiously diluted with MeOH at 50° yielded 2-2.5 mg. IV long plates, m. 204° (under the microscope single crystals were pale purple), sparingly soluble in hexane, more soluble in C₆H₆, insol. in MeOH; when partitioned between hexane and 95% MeOH, the IV is found entirely in the epiphase; mol. extinction coefficient at 487 m μ in hexane, Emol.1 cm. 16.6 + 104. IV (5.269 mg.) in 1:1 cyclohexane-glacial AcOH hydrogenated over 11.5 mg. PtO₂ absorbed 2.76 cc. H (0°, 760 mm.), corresponding to 10.6 double bonds. IV catalyzed with iodine in light gave the 3 main cis-forms (neo A, B, and C), which can be separated from VI by

developing on lime-celite with 1:3 C₆H₆-hexane. The visually observed maximum in hexane of the rechromatographed compds. are all-trans-IV, 521, 490, 457 mμ; VII, 518, 485, 456 mμ; neo B, 514, 483 mμ; neo C, 510, 481 mμ. The C₆H₆-hexane solution of V evaporated and the residue recrystd. from C₆H₆-MeOH gave 1 mg. pure V, dark purple crystals, m. 193°; a total of 14 mg. was obtained from parallel runs; single crystals were purple with a brownish tinge; it was insol. in MeOH, but more soluble in hexane and C₆H₆ than IV, showed epiphasic behavior when partitioned between hexane and 95% MeOH, showed in hexane visual absorption at 534 and 499 mμ; mol. extinction coefficient in hexane at 499 mμ, Emol.1 cm.. 16.0 + 104. The mol. extinction curves of all-trans-IV in fresh hexane, after refluxing 1 hr. and after iodine catalysis in light, of cis-IV, neo A, neo B, neo C, of V, all-trans, neo A, and neo B, in hexane are recorded.

AN 1954:67992 CAPLUS Full-text
DN 48:67992
OREF 48:12042a-g
TI Action of N-bromosuccinimide on β-carotene
AU Zechmeister, L.; Wallcave, L.
CS California Inst. of Technol., Pasadena
SO Journal of the American Chemical Society (1953), 75,
4493-5
CODEN: JACSAT; ISSN: 0002-7863
DT Journal
LA Unavailable

Use Policies apply.

They are available for your review at:

<http://www.cas.org/legal/infopolicy.html>

=> s (1949 and 71 and 1395)/so
53592 1949/SO
275114 71/SO
4222 1395/SO
L6 1 (1949 AND 71 AND 1395)/SO

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L6 ANSWER 1 OF 1 CAPLUS COPYRIGHT 2008 ACS on STN
AB cf. C.A. 43, 4218a. Distn. of 30 g. of brown gummy residue from the fractionation of CH₂:CHCH:CHCN (I) in a short-path apparatus gave 22 g. heavy yellow oil, b0.2 122-3°, nD₂₀ 1.5170. The twice-distilled dimer was treated with Darco in hot 95% EtOH, cooled to 50°, and H₂O added to cloudiness; cooling slowly gave colorless crystals of 1-(2-cyanovinyl)-2- cyano-3-cyclohexene (II), m. 53-4° (from Et₂O-ligroin). II 2.0 and N-bromosuccinimide 2.4 g. in C₆H₆ 45 cc. were refluxed 3 hrs. (neg. test with acid KI for active Br), the solution cooled to 0°, the 1.19 g. precipitated succinimide filtered, the residue from the C₆H₆ filtrate heated with 8 cc. isoquinoline in an oil-bath at 180° 25 min. and at 190° 5 min., cooled, dilute HCl added, and the C₆H₆ extract distilled to give 1.5 g. o-cyanohydrocinnamitrile (III), b0.2 110°, nD₂₀ 1.5468, m. 41-2.5° [from Et₂O-ligroin (b. 30-60°)]. Hydrolysis of 0.5 g. III with 5 cc. 20% KOH and 5 cc. EtOH, refluxing 24 hrs. (cessation of NH₃ evolution), concentration, and addition of concentrated HCl gave 0.12 g. brown insol. base, m. 205-15° (decomposition), which was not identified. Acidification of the filtrate gave 0.07 g. of an infusible neutral solid (diamide of IV?) and, after standing for 16 hrs. 0.28 g. o-HO₂CC₆H₄CH₂CH₂CO₂H (IV), m. 166.5-8° (from H₂O), and same mixed m.p. with authentic IV prepared from 1, 2-dihydronaphthalene and KMnO₄ (cf. C.A. 7, 1499). Higher-boiling I

(nD20 1.4960) 3.0 and picric acid 0.1 g. were heated 16 hrs. on a steambath and distilled to give 2.0 g. oily dimer, nD20 1.5172, and 0.5 g. monomer. Similarly 3.0 g. low-boiling I (nD20 1.4852) gave 0.4 g. dimer, nD20 1.5112, 0.5 g. recovered monomer, and the rest as polymer. Several grams of once-crystallized II, heated with picric acid on the steam bath 25 hrs., and then distilled gave only II and no polymeric residue.

AN 1949:34135 CAPLUS Full-text

DN 43:34135

OREF 43:6172e-i

TI 1-Cyano-1,3-butadiene. III. The dimer of 1-cyano-1,3-butadiene

AU Snyder, H. R.; Poos, George L.

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DT Journal

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